Reference system and centralised calibration for milk (payment) testing

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Outline

- PreCalibration
- Homogenizer Performance Evaluation
- Calibration Samples
- Research to Improve Accuracy of Infrared Milk Analysis

PreCalibration (monthly)

Key Parameters

- Flow system check
- Homogenization efficiency
  evaluated by particle size analysis
- Water and milk repeatability
- Primary slope for each component
- Purging efficiency
- Linearity
  (evaluated with modified milk samples)
- Intercorrection values
  (evaluated with modified milk samples)

Outline

- PreCalibration
- Homogenizer Performance Evaluation

Homogenization Efficiency Testing (monthly)

Three vials pasteurized, unhomogenized milk are sent from Cornell to each lab per instrument each month.

The milk is warmed to 42^{o}C, pumped through the instrument and the instrument homogenized is collected from the by-pass outlet, immediately cooled, and shipped back to Cornell. Each samples is test by laser light scattering to determine the fat globule size distribution. We recommend that a lab replace the homogenizer when the the d(0.9) of the particle size distribution reaches 1.7 microns.

Homogenization Efficiency Testing (monthly)

Recently, we have also started investigating why homogenizers fail. Laboratories send the failed homogenizer to Cornell and we disassemble the homogenizer. We conduct a microscopic examination of the internal parts to try to determine the cause of the homogenizer failure.

Also, when possible, we check the performance of new homogenizers before they are installed on an instrument.

Primary Slope Control (monthly)

When primary slope (i.e., gain) of the primary signal for each measured component is set in a one to one relationship with the change in concentration of that component, the intecorrection factors from one instrument the next become almost identical, particularly among FTIR instruments run in traditional filter mode.

Outline

- PreCalibration
- Homogenizer Performance Evaluation
- Calibration Samples
  - Production of modified milk samples
  - All lab mean chemistry reference values
  - Chemistry method proficiency testing and trouble shooting.
  - Stability of instrument performance and slope intercept values.

Production of Modifed Milk Calibration Samples

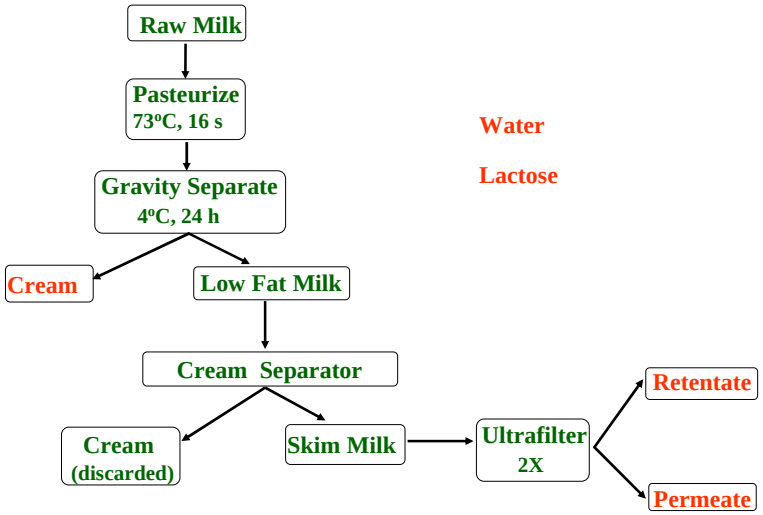
14 milks - an orthogonal matrix of composition with no correlation among component concentrations.

- Fat range = 0.2 to 5.8%
- True protein range = 2 to 4.3%
- Anhydrous lactose range = 3.9 to 5.2%

These samples are used to set slope and intercept of the intercorrected mid-IR signal.

One calibration can be used for raw milk payment testing and for testing of homogenized HTST pasteurized milks (0.2 to 3.6% fat).

Production of Ingredients for Milk Calibration Samples



Formulation of Calibration Samples

- The composition of each ingredient is entered into an Excel spreadsheet.
- The target composition of each of the 14 samples is pre-set in the spreadsheet.
- The optimization solver function of Excel is used to calculate the amount of each ingredient needed for each of the 14 samples to achieve the compositions targets.
- Currently, originally the samples were preserved with potassium dichromate, currently the samples are preserved with Microtabs II (bronopol and delvocid) and have a refrigerated shelf-life of 1 month.

Example: Modified Milk Calibration
Sample Set – June 2, 2008

Sample	Fat	Protein	Solids	Lactose
1	0.2115	4.2463	9.5861	4.0373
2	0.6432	2.2219	8.4074	4.5166
3	1.1157	3.9037	11.3312	5.1119
4	1.5164	2.5634	10.1098	4.9405
5	1.9464	3.5745	10.9290	4.3015
6	2.3774	2.9012	10.9171	4.5483
7	2.8082	3.2422	11.7286	4.5522
8	3.2425	3.0787	11.8243	4.4113
9	3.6722	3.4097	12.9167	4.6744
10	4.1084	2.7470	11.9975	4.1303
11	4.5460	3.7498	14.3034	4.8308
12	4.9743	2.4132	12.3422	3.9908
13	5.4067	4.1000	15.2816	4.5721
14	5.8312	2.0783	14.0166	5.0522
Mean	**3.0286**	**3.1593**	**11.8351**	**4.5479**
min	**0.2115**	**2.0783**	**8.4074**	**3.9908**
max	**5.8312**	**4.2463**	**15.2816**	**5.1119**
range	**5.6197**	**2.1681**	**6.8742**	**1.1211**

14 Modified Milk Samples (Three Purposes)

First, each month the testing of these samples provides a proficiency test of the fat by ether extraction, the true protein by Kjeldahl, the anhydrous lactose by enzymatic, and total solids by oven drying methods. The orthogonal matrix of composition of the set of samples provides some interesting diagnostic and trouble shooting opportunities that are used to improve the performance of the laboratories that run the chemistry methods.

The performance of individual laboratories and the group of laboratories for the chemistry methods has been improved.

14 Modified Milk Samples (Three Purposes)

Second, the all-laboratory mean with outliers removed is used to create a fat, protein, and lactose reference value for each sample.

Third, the samples are used for 1 month to set the slope and intercepts for each instrument. Because of the orthogonal matrix of composition, the data can be used to evaluate and adjust the linearity and intercorrection response of each instrument. These evaluation calculations and protocols are built into a software package we have written called IR-QC.

14 Modified Milk Samples

Instrument Calibration Performance:

Standard Deviation of the Difference (SDD) between Reference Chemistry and Instrument Predictions

Before we started using the modified milks, the SDD with producer calibration samples generally were never less than 0.025% for any component.

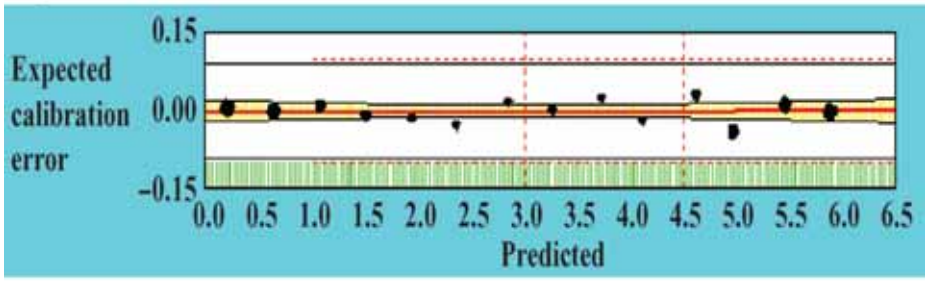
14 Modified Milk Samples

Instrument Calibration Performance:

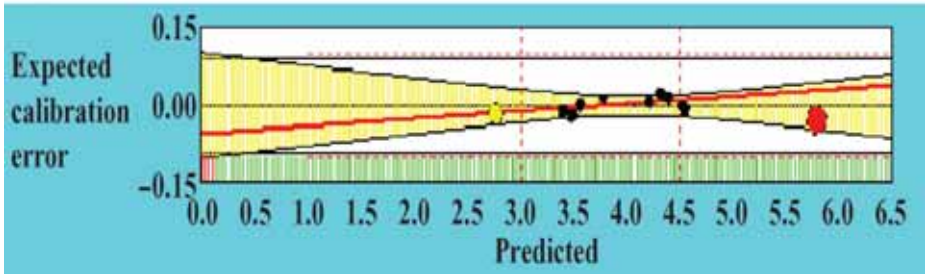
With Modified Milks and all-lab mean reference values, the SDD on all components is < 0.015% and often < 0.01%.

The size of the 95% confidence interval around the slope of the regression line has been reduced greatly by the use of the modified milk calibration samples.

a) Modified Milk Calibration – Fat B



b) Producer Milk Calibration – Fat B



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Research to Improve Accuracy of Infrared Milk Analysis

- Development of an optimized set of traditional “virtual” sample and reference filter wavelengths for use in FTIR instruments. – status: complete and in process of publication.

Research to Improve Accuracy of Infrared Milk Analysis

- Quantitative determination of the impact of variation in fatty chain length and unsaturation on Fat B and Fat A on absorbance at sample and reference wavelengths with a model sample system. – status: complete and in the process of publication.
- Verification of the chain length and unsaturation impacts with producer samples. – status: complete and in the process of publication.

Research to Improve Accuracy of Infrared Milk Analysis

- Develop an improved traditional “virtual filter” calibration approach that minimizes the impact of variation in fatty acid chain length and unsaturation. - status: work in progress.

Research to Improve Accuracy of Infrared Milk Analysis

- Determine the impact of various preservatives on infrared uncorrected signals initially and during calibration sample shelf-life – status: data collection is complete.
- Develop a set of unpreserved modified milk samples that have a refrigerated shelf life of 1 month – status work in progress with some success.

Research to Improve Accuracy of Infrared Milk Analysis

- Continue to implement and apply new statistical quality control tools in IR-QC to calibration data to improve the accuracy of milk testing.

Acknowledgments

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